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Indian Standard

METHOD FOR THE DETERMINATION OF MANGANESE BY ATOMIC ABSORPTION SPECTROPHOTOMETRY

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NEW DELHI 110002

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Indian Standard

METHOD FOR THE DETERMINATION OF MANGANESE BY ATOMIC ABSORPTION SPECTROPHOTOMETER

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Indian Standard

METHOD FOR THE DETERMINATION OF MANGANESE BY ATOMIC ABSORPTION SPECTROPHOTOMETRY

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 16 February 1987, after the draft finalized by the Chemical Standards Sectional Committee had been approved by the Chemical Division Council.

0.2 The atomic absorption spectrophotometric method is dependent on the fact that atoms, in the ground state, will absorb light of the same wavelength they emit when excited. When radiation from given excited atoms is passed through a flame containing atom in the ground state, the intensity of the transmitted radiation will decrease in proportion to the amount of the ground state atoms in the flame. A hollow cathode lamp for the element to be determined or a suitable electrode less discharge lamp provides the radiation. The atoms of the elements to be measured are placed in the beam of radiation by aspirating the specimen into an oxidant fuel flame. The absorbance is taken as a measure of concentration of the analysed atoms.

0.3 Various products where manganese estimation may be required include alloys, high purity metals, edible oils, wort, beer, yeast, rubber chemicals, rubber products, water samples, petroleum products, soils, catalysts, etc.

0.4 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

1. SCOPE

1.1 This standard prescribes the atomic absorption spectrophotometric method for the determination of manganese.

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*Rules for rounding off numerical values (*revised*).

2. QUALITY OF REAGENTS

2.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977*) shall be used in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

3. OUTLINE OF THE METHOD

3.0 Manganese in solution can be determined by atomic absorption spectrophotometric procedure using 279·8 nm wavelength at 0·2 nm band pass in air-acetylene condition.

4. APPARATUS

4.1 Atomic Absorption Spectrophotometer — Generally with the following parameters.

4.1.1 *Lamp Current* — Depending on the lamp and instrument used.

4.1.2 *Support* — Air.

4.1.3 *Fuel* — Acetylene.

4.1.4 *Flame Stoichiometry* — Oxidizing.

4.1.5 *Wavelength and Working Range*

Wavelength, nm	*Band Pass*, nm	*Working Range*
279·8	0·2	0·1-5 μg/ml
403·1	0·2	5-15 μg/ml

NOTE — Rotate the burner position to accommodate high concentration of manganese in the solution.

5. REAGENT

5.1 Manganese Metal — 99·99 percent.

(NOTE — Equivalent quantity of suitable G.R. grade manganese salt may also be taken).

5.2 Dilute Hydrochloric Acid — 1 : 1 (*v/v*).

5.3 Dilute Nitric Acid — 1 percent.

5.4 Stock Standard Solution — Dissolve 0·250 0 g of pure manganese metal (or equivalent weight of suitable G.R. grade manganese-salt) in a minimum volume of (1·1) nitric acid. Dilute to 250 ml volume with

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*Specification for water for general laboratory use (*second revision*).

1 percent hydrochloric acid. This will give 1 000 μg/ml of manganese solution (A). From the solution (A) prepare 100 μg/ml (B) and then prepare 1, 2, 3, 4, 5, 10 μg/ml of manganese solution by appropriate diluting the (B) and adding 2·3 ml of (1:1) hydrochloric acid.

6. PROCEDURE

6.1 Operation — Aspirate the standard solution into the flame and record the corresponding absorbances. Plot the absorbances against the concentrations to get a calibration curve. The unknown concentrations are obtained from the absorbances of the unknown solution and comparing with the linear portion of the calibration curve. The working range is linear up to concentration of 5 μg/ml in aqueous solution for the most sensitive spectral line. Alternatively, the concentration may be obtained by setting the instrument for direct reading of concentration where the apparatus has in-built Microprocessor unit or is coupled with DCR unit.

NOTE 1 — Single element hollow cathode lamp is preferred over the multi-element hollow cathode lamp.

NOTE 2 — It is always preferable to use more sensitive resonance wavelength than the other alternative lines.

NOTE 3 — For measuring higher concentrations of the element rotate the burner or dilute the solution appropriately or measure at less sensitive non-interfering lines.

NOTE 4 — Always use linear portion of the calibration curve for obtaining concentration, if necessary, use curve calibration.

6.2 Calculation — Manganese, percent by mass $= \frac{C \times V}{10^6} \times \frac{100}{M}$

where

C = concentration of manganese in μg/ml in the final solution,
V = volume in ml of the final solution, and
M = mass in g of the sample in the final solution.

INTERNATIONAL SYSTEM OF UNITS (SI UNITS)

Base Units

Quantity	*Unit*	*Symbol*
Length	metre	m
Mass	kilogram	kg
Time	second	s
Electric current	ampere	A
Thermodynamic temperature	kelvin	K
Luminous intensity	candela	cd
Amount of substance	mole	mol

Supplementary Units

Quantity	*Unit*	*Symbol*
Plane angle	radian	rad
Solid angle	steradian	sr

Derived Units

Quantity	*Unit*	*Symbol*	*Definition*
Force	newton	N	1 N = 1 kg,m/s^2
Energy	joule	J	1 J = 1 N.m
Power	watt	W	1 W = 1 J/s
Flux	weber	Wb	1 Wb = 1 V.s
Flux density	tesla	T	1 T = 1 Wb/m^2
Frequency	hertz	Hz	1 Hz = 1 c/s(s^{-1})
Electric conductance	siemens	S	1 S = 1 A/V
Electromotive force	volt	V	1 V = 1 W/A
Pressure, stress	pascal	Pa	1 Pa = 1 N/m^2